

ANALYST:		VPDES NO	
----------	--	----------	--

Parameter: Nitrate-Nitrite Nitrogen

Method: Cadmium Reduction

04/02

METHOD OF ANALYSIS:

	18th Edition of Standard Methods 4500-NO ₃ E
	EPA Methods For Chemical Analysis 353.3
	ASTM D3867-90(B)

		Y	N
1)	Is the cadmium reduction method used for samples with NO ₃ -N NO ₂ -N concentrations ranging from 0.01 mg/L to 1.0 mg/L? [SM-1.a; 353.3-1.1]		
2)	Are samples containing NO ₃ -N NO ₂ -N concentrations greater than 1.0 mg/L diluted to fall within the desired range? [Permit]		
3)	Is the cadmium reduction column prepared as described in the method? [SM-2.a; 353.3-5.1]		
4)	Is the cadmium in the column stored either under distilled water or NH ₄ Cl EDTA solution? [SM-4.b.3; 353.3-6.3]		
5)	Is absorbance measured with a spectrophotometer or filter photometer at 540 nm with a light path of 1.0 cm or longer? [SM-2.b.1; 353.3-5.2]		
6)	Are matched cuvettes used for colorimetric analysis? [Permit]		
7)	Are cuvettes free of fingerprints, scratches or stains? [Permit]		
8)	Are reagents prepared as described in the method? [SM-3; 354.3-6]		
9)	Are all reagents and standards prepared with nitrite - nitrate free distilled water? [SM-3.a; Permit]		
10)	Is the buffer-colored reagent prepared monthly? [SM-3.c; Permit]		
11)	Is the buffer-colored reagent stored in a dark colored bottle at 4°C? [SM-3.c; Permit]		
12)	Is the anhydrous sodium nitrite desiccated for at least 24 hours prior to preparation of the stock nitrite solution? [SM-3.j; Permit]		
13)	Is the stock nitrite solution standardized as described in the 18th Edition of Standard Methods? [SM-3.j; Permit]		
14)	Is the stock nitrite solution prepared quarterly? [Permit; 353.3-6.14]		
15)	Is stock nitrate solution prepared semiannually? [SM 4500-NO ₃ B.3b; 353.3-6.12]		
16)	Is the stock nitrate and nitrite solution preserved with 1 - 2 mL of chloroform per liter and stored a 4°C? [SM 4500-NO ₃ B.3.b/ NO ₂ B.3.e.1; 353.3-6.12/14]		
17)	Are the intermediate and standard nitrite solutions prepared daily? [SM-4500 NO ₂ B.3.f/g; Permit]		
18)	Is intermediate nitrate solution preserved with 1 - 2 mL of chloroform per liter? [SM NO ₃ B.3.c]		
19)	Are standards prepared using Class A glassware? [SM 1070 B.2; Permit]		

NO₂-NO₃

		Y	N
20)	Are samples adjusted to have a pH between 5 and 9 with 1N HCl or NH ₄ OH as required? [SM-4.b.2; 353.3-7.3]		
21)	Are turbid samples filtered through a .45 micron membrane filter or a glass fiber filter? [SM-A.1; 353.3-7.1.1]		
22)	Are excessively turbid samples pretreated with ZnSO ₄ before filtration? [353.3-7.1.2]		
23)	Are samples containing oil and grease extracted with two 25 mL portions of nonpolar solvent (hexane, chloroform)? [SM-1.b; 353.3-7.2]		
24)	Are samples containing residual chlorine dechlorinated with sodium thiosulfate? [SM-1.b; Permit]		
25)	Is a 25 mL sample or an aliquot diluted to 25 mL added to 75 mL of NH ₄ Cl-EDTA solution? [SM-4.b.3; 353.3-7.4]		
26)	Is the first 25 mL of sample discarded from the reduction column and the remainder collected in the sample flask? [SM-4.b.3; 353.3-7.6]		
27)	Is the reduction completed in 10 to 14.5 minutes? [SM-4.b.3; 353.3-7.5]		
28)	Is the buffer-color reagent added within 15 minutes of completion of sample reduction? [SM-4.b.4; 353.3-7.6]		
29)	Are 2 mL of buffer-color reagent added to 50 mL of reduced sample or an aliquot diluted to 50 mL? [SM-4.b.4; 353.3-7.7]		
30)	Is color allowed to develop for a minimum of 10 minutes but no longer than 2 hours before reading absorbance? [SM-4.b.4; 353.3-7.7]		
31)	Are the 5 NO ₃ standards used to prepare the calibration curve carried through the reduction process? [SM-4.c; 353.3-6.16 & 7.8]		
32)	Is a distilled water-reagent blank used to zero the spectrophotometer/photometer? [Permit; 353.3-6.16]		
33)	In order to verify an existing curve, are two standards that bracket the sample values and a reagent blank run with each series of samples with recovery of "10% of known values? If not, is a new curve developed? [Permit]		
35)	Is at least one NO ₂ standard compared to a reduced NO ₃ standard to verify the reduction column efficiency? [SM-4.c; 353.3-7.8]		
36)	Are Cu-Cd granules reactivated when reduction efficiency falls below 75%? [SM-4.c; Permit]		

PROBLEMS: